

PTO 09-7417

CC=JP DATE=19941021 KIND=A  
PN=06292544

METHOD FOR PREPARING FAT-CONTAINING BEVERAGES PACKED IN HERMETIC  
CONTAINERS  
[Mippu Yoki Iri Yushi Gan'yu Inryo No Seiho]

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UNITED STATES PATENT AND TRADEMARK OFFICE  
Washington, D.C. August 2009

Translated by: FLS, Inc.

PUBLICATION COUNTRY	(19): JP
DOCUMENT NUMBER	(11): 06292544
DOCUMENT KIND	(12): A
	(13): PUBLISHED UNEXAMINED APPLICATION (Kokai)
PUBLICATION DATE	(43): 19941021
PUBLICATION DATE	(45):
APPLICATION NUMBER	(21): 05268232
APPLICATION DATE	(22): 19930929
INTERNATIONAL CLASSIFICATION	(51): A23L 2/00; A23C 0/152; A23L 2/38
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TITLE	(54): METHOD FOR PREPARING FAT- CONTAINING BEVERAGES PACKED IN HERMETIC CONTAINERS
FOREIGN TITLE	[54A]:MIPPU YOKI IRI YUSHI GAN'YU INRYO NO SEIHO

[Claim 1] A method for preparing fat-containing beverages packed in hermetic containers, said method having the following process steps in succession:

(1) a step of mixing a fat component, water, and an emulsifier or a stabilizer, thereby forming them into a fat-containing solution having a temperature of 85 °C or lower;

(2) a step of homogenizing the aforesaid fat-containing solution with a pressure of 500 kg/cm<sup>2</sup> or higher, thereby obtaining a homogenized fat-containing solution in which the particle size of the water-insoluble component is 1.0 µm or smaller; and

(3) a step of mixing the aforesaid homogenized fat-containing solution and a solution of an emulsifier or a stabilizer and subsequently packing the mixture in a container and sealing and sterilizing it.

[Claim 2] A method for preparing fat-containing beverages packed in hermetic containers, said method having the following process steps in succession:

(1) a step of mixing a fat component, water, and an emulsifier or a stabilizer, thereby forming them into a fat-containing solution having a temperature of 85 °C or lower;

(2) a step of homogenizing the aforesaid fat-containing solution with a pressure of 500 kg/cm<sup>2</sup> or higher, thereby obtaining a

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\* Claim and paragraph numbers correspond to those in the foreign text.

homogenized fat-containing solution in which the particle size of the water-insoluble component is 1.0  $\mu\text{m}$  or smaller;

(3) a step of cooling the aforesaid homogenized fat-containing solution to 40 °C or lower so as to make it a stably-homogenized, fat-containing solution; and

(4) a step of packing the aforesaid stably-homogenized, fat-containing solution in a container and sealing and sterilizing it.

[Detailed Description of the Invention]

[0001] [Field of Industrial Application]

The present invention relates to a method for producing hermetic container-packed, fat-containing beverages that can maintain a stably emulsified condition in long-term storage even though they contain fat components.

[0002] [Related Art]

Milk beverages, such as milk coffee, milk tea, soup, and the like, cocoa drinks, and so forth are well known as fat-containing beverages that contain fat components, such as milk fat, cacao fat, vegetable oil, and the like. These fat-containing beverages, in the case of producing them as hermetic container-packed beverages to be distributed at room temperature over a long period of time, are usually produced by dissolving the aforesaid fat component and other raw materials in warm water, by homogenizing them with the application of a pressure of from 100 to 250  $\text{kg}/\text{cm}^2$  or thereabouts, and subsequently by packing the homogenized product in containers, such as

cans or the like, and by sealing and sterilizing the product. However, when the aforesaid hermetic container-packed, fat-containing beverages are stored for a long period of time, the fat component floats to the surface of the beverage liquid and forms a ring-shaped cream layer, thus adversely affecting the appearance and decreasing the quality of the beverages.

[0003] Accordingly, to prevent the occurrence of the cream layer, various kinds of emulsifiers, such as sucrose fatty acid esters, organic acid monoglycerol fatty acid esters, polyglycerol fatty acid esters, and the like, are added individually or in combination of several kinds. However, the addition of the aforesaid emulsifiers in order to prevent the occurrence of the cream layer is effective only for beverages whose fat content is 1 % by weight (hereinafter expressed as "%") or less, and, for beverages having a higher fat content, the emulsifiers can hardly be expected to yield the intended effect. In addition, because the addition quantity of the emulsifiers increases as the fat content increases, the bitterness specific to the emulsifiers destroys the flavor of the beverages, thus presenting another problem. Further, when a fresh cream or cacao fat is used as the fat component, the emulsified condition is very likely to become unstable, and a ring layer is formed even when the fat content is 1 % or less, or refrigerated storage causes the fat component to aggregate and form coagulation. Because this coagulation cannot be dispersed

again even by the shaking of the container, it is a major cause of quality deterioration.

[0004] Accordingly, to solve the aforesaid problems, the method described in JP-A-S50-132160, for example, has been proposed. According to this method, an aqueous solution prepared by adding an emulsion/dispersion stabilizer, such as vegetable gum or the like, to powdered cocoa is heated to 60 °C or higher, and an organic acid is added thereto to adjust the pH of the solution to from 5.0 to 5.5 and thereby to cause protein denaturation, after which the solution is homogenized under a pressure of 50 kg/cm<sup>2</sup> or higher and diluted, thereby forming a chocolate drink. With this method, however, when the aqueous solution is at the isoelectric point of protein, the protein denaturation progresses, and the protective colloid effect that the protein itself has is lost. Even if the pH is raised again, the protective colloid effect cannot be obtained; therefore, it becomes necessary to add an emulsifier or stabilizer excessively in order to maintain the fat in an emulsified state.

[0005] As another method to solve the aforesaid problems, there is the method described in JP-B-H02-059708. This method is a method for producing high-fat-content soymilk, according to which, after a vegetable oil and emulsifier are added to a ground soybean liquid, they are stirred at from 95 to 100 °C for from 3 to 10 minutes and subsequently homogenized under a pressure of from 400 to 1000 kg/cm<sup>2</sup>. According to this method, however, the fat-containing solution is

stirred at a high temperature of 95 °C or higher and then homogenized. Therefore, in the high-pressure homogenization process, the temperature of the fat-containing solution further rises due to the collision of particles and ends up reaching 110 °C or higher. The homogenized solution, which has had such a high-temperature heat history, undergoes a higher-temperature heat history for a long time in the subsequent sealing and sterilizing process, and the proteins are thermally denatured and form aggregates or the flavor is destroyed, thus presenting problems.

[0006] As yet another method to solve the aforesaid problems, there is the method described in JP-A-H01-252273. According to this method, glycerol fatty acid ester and iota carrageenan are added to and dissolved in a raw-material liquid comprised of a fat-containing milk component and pre-emulsified with a high-speed agitator, after which the liquid is homogenized under a pressure of from 200 to 250 kg/cm<sup>2</sup> and combined with a coffee extract or fruit juice and a sweet constituent, and the obtained mixture is heated and sterilized and also homogenized again. With this method, however, after the heat sterilization, the product is homogenized again and packed in containers; therefore, unless (1) the product is packed in containers in a sterile condition by sterilizing the containers in advance or (2) the product is packed in containers and then sterilized again, the product cannot be kept for a long period of time, but conducting such processes makes the sterilization process troublesome, or the product

undergoes the sterilization process twice and experiences an excess heat history, thus presenting the problem of impairing the flavor of the beverage. In addition, since this method employs a low homogenization pressure of from 200 to 250 kg/cm<sup>2</sup>, it cannot fully emulsify a high-fat-content beverage whose fat content exceeds 1 %.

[0007] [Problems that the Invention Intends to Solve]

The present invention was achieved in view of this situation, and its objective is to provide a method for producing hermetic container-packed, fat-containing beverages that, without undergoing an excess heat history, can maintain a stably emulsified condition over a long period of time even if they contain fat components at high concentrations.

[0008] [Means for Solving the Problems]

The aforesaid objective can be attained by the hermetic container-packed, fat-containing beverage production method that has the following process steps in succession:

(1) a step of mixing a fat component, water, and an emulsifier or a stabilizer, thereby forming them into a fat-containing solution having a temperature of 85 °C or lower;

(2) a step of homogenizing the aforesaid fat-containing solution with a pressure of 500 kg/cm<sup>2</sup> or higher, thereby obtaining a homogenized fat-containing solution in which the particle size of the water-insoluble component is 1.0 μm or smaller; and



(3) a step of mixing the aforesaid homogenized fat-containing solution and a solution of an emulsifier or a stabilizer and subsequently packing the mixture in a container and sealing and sterilizing it.

[0009] The aforesaid objective can also be attained by the hermetic container-packed, fat-containing beverage production method that has the following process steps in succession:

(1) a step of mixing a fat component, water, and an emulsifier or a stabilizer, thereby forming them into a fat-containing solution having a temperature of 85 °C or lower;

(2) a step of homogenizing the aforesaid fat-containing solution with a pressure of 500 kg/cm<sup>2</sup> or higher, thereby obtaining a homogenized fat-containing solution in which the particle size of the water-insoluble component is 1.0 μm or smaller;

(3) a step of cooling the aforesaid homogenized fat-containing solution to 40 °C or lower so as to make it a stably-homogenized, fat-containing solution; and

(4) a step of packing the aforesaid stably-homogenized, fat-containing solution in a container and sealing and sterilizing it.

[0010] That is, for the purpose of stabilizing the emulsified state of high-fat-content beverages for a long period time, the present inventors conducted research firstly on the conditions of the solution to be homogenized. As a result, they learned that emulsion stability is improved by homogenizing a fat-containing solution

together with an emulsifier or stabilizer, subsequently by further adding a solution containing an emulsifier or stabilizer thereto, and by packing the resulting product in containers and sealing and sterilizing the product, or, alternatively, by cooling the solution after the homogenization, followed by packing in containers and sealing and sterilizing them. Based on this finding, the present invention was achieved.

[0011] The following will explain the present invention in detail. Examples of the fat-containing beverages pertaining to the present invention include milk coffee, Viennese-style coffee, milk tea, cocoa, chocolate drinks, soup, miso soup, milk-red bean soup, milk shake, powdered green tea milk, acidic milk beverages, and so forth. Examples of the fat components used in the aforesaid fat-containing beverages include: fresh cream, milk, whole powdered milk, condensed milk, soy milk [sic], butter, cheese, and the like, which contain milk fats derived from dairy products; cacao fat, cacao-equivalent fat, vegetable oil, coconut oil, margarine, medium-chain triglyceride, nuts paste, and the like, which contain vegetable fats, and processed products thereof; fish oil, lard, tallow, egg oil, and the like, which contain animal fats, and processed products thereof; and so forth. These may be used singly or in combination of two or more kinds according to the objective.

[0012] Next, one of the present invention's methods for producing hermetic container-packed, fat-containing beverages is carried out as

follows. That is, as the first step, water is added to and mixed with the aforesaid fat components and an emulsifier or a stabilizer, thereby forming a fat-containing solution having a temperature of 85 °C or lower. Here, it is desirable to set the solid content in the fat-containing solution to from 8 to 50 % for achieving a better homogenization effect in the subsequent step. The fat content is set to from 0.005 to 30 % according to the objective. It is highly preferable to prepare the fat-containing solution in such a manner that the final-product, fat-containing beverage will have a fat content of 0.5 % or more, better yet, 1 % or more.

[0013] The stabilizer or emulsifier used in the present invention may be any that has been used heretofore. Examples of the stabilizers include: seaweed extracts, such as carrageenan, agar, alginic acid, sodium alginate, and the like; viscous substances derived from plants or microorganisms, such as guar gum, pectin, gum arabic, curdlan, xanthan gum, locust bean gum, carboxymethyl cellulose, and the like; and proteins, such as gelatin, egg white, casein sodium, and the like.

[0014] Examples of the emulsifiers include monoglycerol fatty acid esters, sucrose fatty acid esters, organic acid monoglycerol fatty acid esters, polyglycerol fatty acid esters, sorbitan fatty acid esters, propylene glycol fatty acid esters, lecithin, and the like. The present invention may use either the emulsifiers or stabilizers or both concomitantly. Furthermore, the present invention may use one type of stabilizer or emulsifier or various kinds in combination. The

addition quantity of the emulsifier or stabilizer in this step is preferably set to from 0.005 to 0.3 % of the weight of the whole fat-containing solution for better emulsion stability.

[0015] As other ingredients, the fat-containing beverages may further incorporate saccharides, nonfat dry milk, nonfat condensed milk, processed products of fruits, vegetables, and the like, sour agents, salts, spices, coloring agents, starches, seasonings, and the like, which are selected as appropriate according to the need.

[0016] The temperature of the fat-containing solution must be set to 85 °C or lower. If it exceeds 85 °C, the temperature of the homogenized fat-containing solution in the later-described homogenization step rises to 90 °C or higher, and the solution is exposed to an excessive heat history, which is likely to cause the fat component and other ingredients in the fat-containing solution to have degradation phenomena, such as thermal denaturation, oxidation, and the like, or the aroma components to be lost, thereby deteriorating the flavor. To adjust the temperature to 85 °C or lower, 95 °C or lower warm water may be used as the water; the temperatures of the fat component and water are adjusted so that the temperature becomes 85 °C or lower when they are blended; or the temperature of the fat-containing solution may be raised as appropriate. The temperature-raising method here may be selected as appropriate from addition of a heated aqueous medium, such as warm water or the like, indirect

heating by the circulation of hot water or steam with the use of a jacket-type tank, heating with a plate-type heater, and so forth.

[0017] Further, in this step, the temperature of the fat-containing solution is preferably set to 40 °C or higher. If the temperature of the fat-containing solution is lower than 40 °C, it becomes difficult to obtain a homogenization effect and consequently to reduce the particle size of the water-insoluble components.

[0018] Next, the aforesaid 85 °C or lower fat-containing solution is homogenized under a pressure of 500 kg/cm<sup>2</sup> or higher, thereby forming a homogenized fat-containing solution whose water-insoluble component has a particle size of 1.0 μm or smaller, preferably 0.7 μm or smaller. Examples of the homogenizer that may be used in this process step are homogenizers that have a mechanism for causing emulsified products to collide with one another or to collide against the vessel wall, such homogenizers including: high-speed homomixers, like those shown in Fig. 2; Manton-Gaulin homogenizers, like the one shown in Fig. 3; homogenizers that have a structure in which the liquid passage branches and subsequently converges, as shown in Fig. 1; and the like.

[0019] As shown in Figs. 2 (a) through (c), high-speed homomixers effect homogenization by the shearing force, impact, collision between particles, and the like that occur while the turbine blades (17) inside the stator (16) are rotated at a high speed and thereby agitate the liquid at a high speed. A Manton-Gaulin homogenizer, as shown in

Fig. 3, feeds a given quantity of a sample (30) into a thin gap (C) between valve 22 and valve 23 with the application of high pressure, which causes the collision of particles, which, in turn, effects homogenization. As the pressure used in a Manton-Gaulin homogenizer, the total pressure applied to the sample (30) before it passes through the gap (C) is detected.

[0020] In Fig. 1, reference numeral 1 indicates a pump; 2, a pressure gauge; 3, a chamber; 4, a liquid-transfer passage; 5a and 5b, split passages (thin pipes); 6, a liquid-transfer passage; and 7, a product outlet port. In this homogenizer, liquid-transfer passage 4 becomes two split passages (5a, 5b) at diverging point A, and these split passages (5a, 5b) converge at converging point B and become liquid-transfer passage (5 [sic]). Owing to this structure, when a sample (10) is fed to liquid-transfer passage 4 by means of the pump (1) with the application of high pressure, the particles inside the sample (10) diverge at the diverging point (A) while colliding with one another, and subsequent passage through the thin flow channel of each split passage (5a, 5b) causes the particles to further collide with one another. Then, the impact caused by the convergence of the liquids from the split passages (5a, 5b) at the converging point (B) thereof causes further collision of particles. As the pressure used in this homogenizer, the pressure applied until the sample (10) enters the split passages (5a, 5b) is detected.

[0021] Therefore, compared with the aforesaid high-speed homomixers and Manton-Gaulin homogenizer shown in Figs. 2 and 3, homogenizing pressure is applied uniformly to the liquid as a whole, and the particle size of the water-insoluble component in the liquid can be made smaller uniformly. Concrete examples of the aforesaid homogenizer include "Nanomizer," a product of Nanomizer Co., "Microfluidizer," a product of Microfluidics Co., and so forth.

[0022] The aforesaid homogenizing pressure is set to  $500 \text{ kg/cm}^2$  or higher, preferably from 700 to  $1500 \text{ kg/cm}^2$ . If the pressure is lower than  $500 \text{ kg/cm}^2$ , insufficient homogenization results, and the particle size of the water-insoluble component is not reduced; as a consequence, a cream layer is formed in long-term storage. Incidentally, the water-insoluble component here means the fat component that has been compounded with a stabilizer or emulsifier and has become water-insoluble. The aforesaid homogenization may be carried out once or repeated two or more times and should be set as appropriate according to the pressure and the type of the fat-containing solution.

[0023] Meanwhile, in addition to the aforesaid homogenized fat-containing solution, a solution in which the aforesaid stabilizer or emulsifier is dissolved is prepared. The aforesaid solution may incorporate, as necessary, the aforesaid other ingredients that are selected as appropriate. For better emulsion stability, the addition quantity of the stabilizer or emulsifier is preferably set to from

0.005 to 0.3 % of the quantity of the final preparation. This solution is mixed with the homogenized fat-containing solution. Here, the proportion of this solution to the homogenized fat-containing solution is preferably 1 (the solution) or less to 5 (the homogenized fat-containing solution) for better emulsion stability. Incidentally, the aforesaid homogenizers may be used for the mixing performed in the step of formulating the aforesaid fat-containing solution or stabilizer- or emulsifier-containing solution or in the step of mixing the aforesaid solutions.

[0024] If an emulsifier, such as a sucrose fatty acid ester, polyglycerol fatty acid ester, or the like, that has a bacteriostatic effect on heat-resistant spore-forming bacteria is used as the emulsifier, it exerts its bacteriostatic activity well and prevents the obtained hermetic container-packed, fat-containing beverage from becoming rancid even if it is stored for a long period, thus maintaining its quality. The effect of an emulsifier, such as a sucrose fatty acid ester or the like, as a bacteriostatic agent is likely to be lost if the homogenization is carried out with the application of a pressure that is  $500 \text{ kg/cm}^2$  or higher because the emulsifier forms an emulsion complex with the fat component. For this reason, if an emulsifier having bacteriostatic activity is added after the homogenization, its effect will not be lost, and a good bacteriostatic effect can be achieved.



[0025] Next, a mixture of the aforesaid solution and homogenized fat-containing solution is packed in containers, such as cans, bottles, paper packs, retort pouches, or the like, sealed, and subjected to retort sterilization. The sealing may be conducted after the sterilization. The hermetic container-packed, fat-containing beverage thus obtained is a beverage that has excellent fat-component emulsion stability and does not form a ring layer derived from the fat component even when stored for a long period, thus having an excellent appearance.

[0026] As the second production method of the present invention, in place of adding, to the aforesaid homogenized solution, a solution in which a stabilizer or emulsifier is dissolved, the homogenized solution may be cooled to 40 °C or lower, preferably to a range of from 5 to 35 °C. If the cooling temperature is higher than 40 °C, the fat component does not become a stable micelle; as a consequence, the emulsion is destroyed by the heat history in the later-described sterilization process, and a cream layer is formed in long-term storage. As the method for effecting the aforesaid cooling, addition of cold water, indirect cooling by the circulation of cold water or a cold medium with the use of a jacket-type tank, cooling with a plate-type cooler, or so forth is used as appropriate.

[0027] Next, the aforesaid homogenized fat-containing solution that has been cooled is directly, or after being heated, packed in containers, such as cans, bottles, paper packs, retort pouches, or the

like, sealed, and subjected to retort sterilization. The sealing may be conducted after the sterilization.

[0028] The hermetic container-packed, fat-containing beverage thus obtained is a beverage that has excellent fat-component emulsion stability and does not form a ring layer derived from the fat component even when stored for a long period, thus having an excellent appearance. Furthermore, the hermetic container-packed, fat-containing beverages obtained by either of the aforesaid production methods may be distributed at ordinary temperature or, alternatively, may be sold in a heated or refrigerated state as necessary. The aforesaid production methods yield approximately the same effect with respect to emulsion stability, but the former production method is preferable from the standpoint of bacteriostatic activity. Furthermore, it is also possible to cool the homogenized solution and subsequently add the solution in which a stabilizer or emulsifier is dissolved, thus combining the aforesaid production methods. Alternatively, after the solution in which a stabilizer or emulsifier is dissolved is added to the homogenized solution, the mixture may be cooled. Here, if an emulsifier having bacteriostatic activity is added, an excellent bacteriostatic characteristic can be imparted to the resulting beverage.

[0029] [Effects of the Invention]

As explained in the foregoing, according to the present invention's methods for producing hermetic container-packed, fat-

containing beverages, the homogenization is conducted with the application of a specific homogenizing pressure, and the homogenized fat-containing solution is mixed with an emulsifier- or stabilizer-containing solution, or the homogenized fat-containing solution is cooled. As a result, beverages containing fats in a high concentration of 1 % or higher and beverages containing fats, such as cacao fat, fresh cream, etc., that are unstable specifically to emulsification, can maintain a stable emulsion state for a long period of time.

[0030] The following will explain the present invention in further detail by presenting working examples.

<Working Examples 1 through 4 and Comparative Example 1>

With the compositions shown in Table 1, homogenized fat-containing solution A and solution B were prepared, after which the entire amounts of these solutions were mixed, thereby obtaining a preparation. The formulation of homogenized fat-containing solution A was carried out under the conditions shown in Table 2, and solution B was prepared by mixing and dissolving the ingredients at 60 °C with a high-speed homomixer.

[0031] [Table 1]

(parts by weight)

脂肪含有均質化溶液A		溶液B	
カカオ豆抽出液 (*1)	2500	砂糖 (f)	550
全粉乳 (a)	650	色素 (g)	10
乳化剤 (*2)	2	乳化剤 (*2)	3
安定剤 (*3)	1	安定剤 (*3)	1
水 (b)	847	食塩 (h)	2
		水 (b)	434
計 (c)	4000	計 (c)	1000
油脂含有量 (重量%) (d)	4.25	油脂含有量 (d)	0
全固形分 (重量%) (e)	16.5	全固形分 (e)	56.6

Key: A) homogenized fat-containing solution; B) solution; \*1) cacao bean extract; \*2) emulsifier; \*3) stabilizer; a) whole powdered milk; b) water; c) total; d) fat content (% by weight); e) total solid content (% by weight); f) sugar; g) colorant; h) table salt.

#### \*1 Cacao extract preparation method

To 10 parts by weight of 90 °C warm water was added 1 part by weight of cacao nibs, which were agitated for 10 minutes, after which the mixture was subjected to solid-liquid separation with a paper filter, and the obtained liquid portion was used as the cacao extract.

\*2 Emulsifier: sucrose fatty acid ester P-1570 (a product of Mitsubishi Kasei Foods Co.)

\*3 Stabilizer: Carrageenan CS409 (a product of San'ei Chemical Co.)

[0032] <Comparative Example 2>

The ingredients of homogenized fat-containing solution A and solution B used in Working Example 1 were mixed together all at once,

and the temperature of the mixture was adjusted to 60 °C. The mixture was then homogenized at 1000 kg/cm<sup>2</sup>.

[0033] (Emulsion stability confirmation test)

Each preparation thus obtained was packed in glass bottles (capacity: 240 cc, diameter: 65 mm, height: 120 mm), and the bottles were sealed, after which they were sterilized by heating at 121 °C for 20 minutes. The bottled beverage thus obtained was left standing at 25 °C, and the presence or absence of a cream layer was visually checked each week. The period required for the cream layer to have a thickness of 1 mm or more was investigated.

(Bacteriostatic property confirmation test)

Each preparation was packed in 190 cc-capacity cans, and, after  $7.0 \times 10^{-4}$  spores of heat-resistant flat sour bacteria were inoculated therein, the can was sealed and sterilized by heating at 121 °C for 20 minutes. The canned beverage thus obtained was stored at 55 °C for one month, and the presence and absence of rancidification was then checked based on the degree of vacuum and pH decrease relative to the uninoculated product. The product was evaluated based on the number of rancidified cans among 10 cans. The results thus obtained are also shown in Table 2.

[0034]

[Table 2]

			實 施 例 (a)				比 較 例 (b)	
			1	2	3	4	1	2
(c) 條 件	昇溫溫度 (°C) (d)		60	60	40	60	60	60
	均質機 (e)		*1	*1	*1	*2	*1	*1
	均質化壓力 (kg/cm <sup>2</sup> ) (f)		1000	700	500	1000	450	1000
	最大粒子徑 (μm) (g)		0.50	0.58	0.68	0.65	1.85	0.82
(h) 評 價 值	乳化安定性 (i) *3		15	12	10	10	1	3
	腐败数 (缶) (k)		0/10	0/10	0/10	0/10	0/10	10/10
	(l) *4 真空度 (mmHg)	未植菌 <sup>(m)</sup>	38	36	36	37	36	37
		植菌後 <sup>(n)</sup>	38	35.9	36	37	36	28.9
	pH	未植菌 <sup>(m)</sup>	6.43	6.45	6.47	6.44	6.45	6.40
		植菌後 <sup>(n)</sup>	6.43	6.44	6.47	6.44	6.45	5.74

Key: a) working examples; b) comparative examples; c) conditions; d) raised temperature; e) homogenizer; f) homogenizing pressure; g) largest particle size; h) evaluation; i) emulsion stability; j) bacteriostatic activity; k) number of rancidified cans; l) degree of vacuum; m) uninoculated; n) after inoculation.

\*1 Nanomizer (a product of Nanomizer Co.)

\*2 Manton-Gaulin

\*3 Period (in weeks) till a cream layer was formed

\*4 Average of 10 cans

[0035] As seen from the results shown in Table 2, the bottled beverages of the working examples did not form any cream layer for 10 weeks or longer. In contrast, the bottled beverages of the comparative examples formed cream layers in 3 weeks or so, thus rendering

themselves undesirable. Furthermore, the bottled beverages of the working examples also exhibited good bacteriostatic activity.

[0036] <Working Examples 5 through 8 and Comparative Examples 3 and 4>

The ingredients shown in Table 3 were mixed and dissolved, and water was added thereto to a prescribed quantity, thereby obtaining a preparation. This preparation was treated under the conditions shown in Table 4 and packed in glass bottles (capacity: 240 cc, diameter: 65 mm, height: 120 mm), and, after the bottles were seamed, they were sterilized by heating at 121 °C for 20 minutes, thereby obtaining a hermetic container-packed, fat-containing beverage. The bottled beverage thus obtained was left standing at 25 °C, and the presence or absence of a cream layer was visually checked each week. The period required for the cream layer to have a thickness of 1 mm or more was investigated. The results are shown in Table 4.

[0037] [Table 3]

カカオ豆抽出液 *1	5000
牛乳 (a)	3000
砂糖 (b)	600
色素 (c)	10
食塩 (d)	2
乳化剤 *2	5
安定剤 *3	2
水 (e)	1381

Key: \*1) cacao bean extract; \*2) emulsifier; \*3) stabilizer; a) milk; b) sugar; c) colorant; d) table salt; e) water.

\*1 Cacao extract preparation method

To 10 parts by weight of 90 °C warm water was added 1 part by weight of cacao nibs, which were agitated for 10 minutes, after which the mixture was subjected to solid-liquid separation with a paper filter, and the obtained liquid portion was used as the cacao extract.

\*2 Emulsifier: sucrose fatty acid ester P-1570 (a product of Mitsubishi Kasei Foods Co.)

\*3 Stabilizer: Carrageenan CS409 (a product of San'ei Chemical Co.)



[0038] [Table 4]

	實施例 (a)				比較例 (b)	
	5	6	7	8	3	4
昇溫溫度 (°C) (c)	60	60	80	60	60	60
均質化壓力 (kg/cm <sup>2</sup> ) *1	500	700	500	1000	700	300
冷卻溫度 (°C) (d)	20	20	40	20	50	20
最大粒子徑 (μm) (e)	0.92	0.68	0.87	0.54	1.27	1.89
乳化安定性 *2	10	12	10	13	2	1

Key: \*1) homogenizing pressure; \*2) emulsion stability; a) working examples; b) comparative examples; c) raised temperature; d) cooled temperature; e) largest particle size.

\*1 Nanomizer (a product of Nanomizer Co.) was used as the homogenizer.

\*2 Period (in weeks) until a cream layer was formed.

[0039] As seen from the results shown in Table 4, the thickness of the cream layers in all of the working examples was 0.2 mm or less, thus indicating a good emulsified state. In contrast, in the comparative examples, cream layers were formed in 2 weeks or so, thus rendering these examples undesirable.

[Brief Explanation of the Drawing]

[Fig. 1] An explanatory drawing illustrating one example of the homogenizer mechanism used in the present invention.

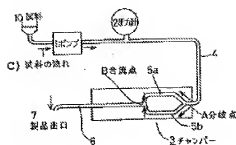
[Fig. 2] An explanatory drawing illustrating examples of the homogenizer mechanism used in the present invention.

[Fig. 3] An explanatory drawing illustrating one example of the homogenizer mechanism used in the present invention.

[Explanation of Reference Numerals]

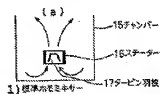
- 1 Pump
- 2 Pressure gauge
- 3 Chamber
- 4 Liquid-transfer passage
- 5 Split passage
- 6 Liquid-transfer passage
- 7 Product outlet port
- 10 Sample

[FIG. 1]

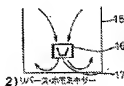


Key: A) diverging point; B) converging point; C) flow of the sample;  
 1) Pump; 2) Pressure gauge; 3) Chamber; 7) Product outlet port; 10)  
 Sample.

[FIG. 2]



(b)

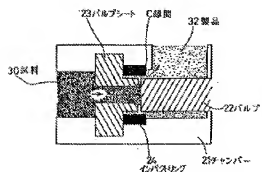


(c)



Key: 1) standard homomixer; 2) reverse homomixer; 3) ultra-mixer; 15)  
 chamber; 16) stator; 17) turbine blade

[FIG. 3]



Key: C) gap; 21) chamber; 22) valve; 23) valve seat; 24) impact ring;  
30) sample; 32) product

[Written Amendment]

[Filing Date] February 7, 1994

[Amendment 1]

[Document to be amended] Specification

[Item to be amended] Claims

[Method of amendment] Conversion

[Details of the amendment]

[Claims]

[Claim 1] A method for preparing fat-containing beverages packed in hermetic containers, said method having the following process steps in succession:

(1) a step of mixing a fat component, water, and an emulsifier or a stabilizer, thereby forming them into a fat-containing solution having a temperature of 85 °C or lower;

(2) a step of homogenizing the aforesaid fat-containing solution, thereby obtaining a homogenized fat-containing solution in which the particle size of the water-insoluble component is 1.0  $\mu\text{m}$  or smaller; and

(3) a step of mixing the aforesaid homogenized fat-containing solution and a solution of an emulsifier or a stabilizer and subsequently packing the mixture in a container and sealing and sterilizing it.

[Claim 2] A method for preparing fat-containing beverages packed in hermetic containers, said method having the following process steps in succession:

(1) a step of mixing a fat component, water, and an emulsifier or a stabilizer, thereby forming them into a fat-containing solution having a temperature of 85 °C or lower;

(2) a step of homogenizing the aforesaid fat-containing solution, thereby obtaining a homogenized fat-containing solution in which the particle size of the water-insoluble component is 1.0 µm or smaller;

(3) a step of cooling the aforesaid homogenized fat-containing solution to 40 °C or lower so as to make it a stably-homogenized, fat-containing solution; and

(4) a step of packing the aforesaid stably-homogenized, fat-containing solution in a container and sealing and sterilizing it.

[Amendment 2]

[Document to be amended] Specification

[Item to be amended] 0006

[Method of amendment] Conversion

[Details of the amendment]

[0006] As yet another method to solve the aforesaid problems, there is the method described in JP-A-H01-252273. According to this method, glycerol fatty acid ester and iota carrageenan are added to and dissolved in a raw-material liquid comprised of a fat-containing milk component and pre-emulsified with a high-speed agitator, after

which the liquid is homogenized under a pressure of from 200 to 250 kg/cm<sup>2</sup> and combined with a coffee extract or fruit juice and a sweet constituent, and the obtained mixture is heated and sterilized and also homogenized again. With this method, however, after the heat sterilization, the product is homogenized again and packed in containers; therefore, unless (1) the product is packed in containers in a sterile condition by sterilizing the containers in advance or (2) the product is packed in containers and then sterilized again, the product cannot be kept for a long period of time, but conducting such processes makes the sterilization process troublesome, or the product undergoes the sterilization process twice and experiences an excess heat history, thus presenting the problem of impairing the flavor of the beverage. In addition, this method cannot fully emulsify a high-fat-content beverage whose fat content exceeds 1 %.

[Amendment 3]

[Document to be amended] Specification

[Item to be amended] 0008

[Method of amendment] Conversion

[Details of the amendment]

[0008] [Means For Solving The Problems]

The aforesaid objective can be attained by the hermetic container-packed, fat-containing beverage production method that has the following process steps in succession:

(1) a step of mixing a fat component, water, and an emulsifier or a stabilizer, thereby forming them into a fat-containing solution having a temperature of 85 °C or lower;

(2) a step of homogenizing the aforesaid fat-containing solution, thereby obtaining a homogenized fat-containing solution in which the particle size of the water-insoluble component is 1.0 μm or smaller; and

(3) a step of mixing the aforesaid homogenized fat-containing solution and a solution of an emulsifier or a stabilizer and subsequently packing the mixture in a container and sealing and sterilizing it.

[Amendment 4]

[Document to be amended] Specification

[Item to be amended] 0009

[Method of amendment] Conversion

[Details of the amendment]

[0009] The aforesaid objective can also be attained by the hermetic container-packed, fat-containing beverage production method that has the following process steps in succession:

(1) a step of mixing a fat component, water, and an emulsifier or a stabilizer, thereby forming them into a fat-containing solution having a temperature of 85 °C or lower;



(2) a step of homogenizing the aforesaid fat-containing solution, thereby obtaining a homogenized fat-containing solution in which the particle size of the water-insoluble component is 1.0  $\mu\text{m}$  or smaller;

(3) a step of cooling the aforesaid homogenized fat-containing solution to 40 °C or lower so as to make it a stably-homogenized, fat-containing solution; and

(4) a step of packing the aforesaid stably-homogenized, fat-containing solution in a container and sealing and sterilizing it.

[Amendment 5]

[Document to be amended] Specification

[Item to be amended] 0014

[Method of amendment] Conversion

[Details of the amendment]

[0014] Examples of the emulsifiers include glycerol fatty acid esters, sucrose fatty acid esters, organic acid monoglycerol fatty acid esters, polyglycerol fatty acid esters, sorbitan fatty acid esters, propylene glycol fatty acid esters, lecithin, and the like. The present invention may use either the emulsifiers or stabilizers or both concomitantly. Furthermore, the present invention may use one type of stabilizer or emulsifier or various kinds in combination. The addition quantity of the emulsifier or stabilizer in this step is preferably set to from 0.005 to 0.3 % of the weight of the whole fat-containing solution for better emulsion stability.

[Amendment 6]

[Document to be amended] Specification

[Item to be amended] 0018

[Method of amendment] Conversion

[Details of the amendment]

[0018] Next, the aforesaid 85 °C or lower fat-containing solution is homogenized, thereby forming a homogenized fat-containing solution whose water-insoluble component has a particle size of 1.0  $\mu\text{m}$  or smaller, preferably 0.7  $\mu\text{m}$  or smaller. If the particle size of the water-insoluble component is larger than 1.0  $\mu\text{m}$ , the water-insoluble component floats up to the liquid surface or precipitates to the bottom; thus, a stably emulsified condition cannot be obtained. Examples of the homogenizer used in the homogenization are homogenizers that have a mechanism for causing emulsified products to collide with one another or to collide against the vessel wall, such homogenizers including: high-speed homomixers, like those shown in Fig. 2; Manton-Gaulin homogenizers, like the one shown in Fig. 3; homogenizers that have a structure in which the liquid passage branches and subsequently converges, as seen in Fig. 1; and the like.

[Amendment 7]

[Document to be amended] Specification

[Item to be amended] 0022

[Method of amendment] Conversion

[Details of the amendment]

[0022] The aforesaid homogenizing pressure is set to preferably 500 kg/cm<sup>2</sup> or higher, better yet, to from 700 to 1500 kg/cm<sup>2</sup>. If the pressure is lower than 500 kg/cm<sup>2</sup>, it becomes difficult to reduce the particle size of the water-insoluble component; as a consequence, a cream layer is likely to be formed in long-term storage. Incidentally, the water-insoluble component here means the fat component that has been compounded with a stabilizer or emulsifier and has become water-insoluble. The aforesaid homogenization may be carried out once or repeated two or more times and should be set as appropriate according to the pressure and the type of the fat-containing solution.

[Amendment 8]

[Document to be amended] Specification

[Item to be amended] 0029

[Method of amendment] Conversion

[Details of the amendment]

[0029] [Effects of the Invention]

As explained in the foregoing, according to the present invention's methods for producing hermetic container-packed, fat-containing beverages, the particle size of the water-insoluble component in the beverages is set to a given size or smaller by homogenization, and the homogenized fat-containing solution is mixed with an emulsifier- or stabilizer-containing solution, or the homogenized fat-containing solution is cooled. As a result, beverages containing fats in a high concentration of 1 % or higher and beverages

containing fats, such as cacao fat, fresh cream, etc., that are unstable specifically to emulsification, can maintain a stable emulsion state for a long period of time. Furthermore, the present invention's methods for producing hermetic container-packed fat-containing beverages can also be applied to fat-containing food products, such as sauces, dressings, dessert mixes, and so forth.

[Amendment 9]

[Document to be amended] Specification

[Item to be amended] 0034

[Method of amendment] Conversion

[Details of the amendment]

[0034] [Table 2]

		実施例 (a)				比較例 (b)	
		1	2	3	4	1	2
(c) 条件	昇温温度 (°C) (d)	60	60	40	60	60	60
	均質機 (e)	*1	*1	*1	*2	*2	*1
	均質化圧力 (kg/cm <sup>2</sup> ) (f)	1000	700	500	1000	150	1000
	最大粒子径 (μm) *3 (g)	0.50	0.58	0.68	0.65	1.35	0.82
(h) 評価値	乳化安定性 (i) *4	15	12	10	10	1	3
	変形数 (倍) (k)	0/10	0/10	0/10	0/10	0/10	10/10
	(j) 静置 真空度 (cmHg)	未植菌 (m)	38	36	36	37	36
		植菌後 (n)	38	35.9	36	37	36
	性 pH *5	未植菌 (m)	6.43	6.45	6.47	6.44	6.45
		植菌後 (n)	6.43	6.44	6.47	6.44	6.45

Key: a) working examples; b) comparative examples; c) conditions; d) raised temperature; e) homogenizer; f) homogenizing pressure; g) largest particle size; h) evaluation; i) emulsion stability; j) bacteriostatic activity; k) number of rancidified cans; l) degree of vacuum; m) uninoculated; n) after inoculation

\*1 Nanomizer (a product of Nanomizer Co.)

\*2 Manton-Gaulin

\*3 The largest particle size of the water-insoluble component in the homogenized fat-containing solution immediately after the homogenization [measured with a laser-type particle size distribution analyzer (SALD-1000), a product of Shimazu Co.]

\*4 Period (in weeks) till a cream layer was formed

\*5 Average of 10 cans

[Amendment 10]

[Document to be amended] Specification

[Item to be amended] 0036

[Method of amendment] Conversion

[Details of the amendment]

[0036] <Working Examples 5 through 8 and Comparative Examples 3 and 4>

The ingredients shown in Table 3 were mixed and dissolved, and water was added thereto to a prescribed quantity, thereby obtaining a preparation. This preparation was treated under the conditions shown in Table 4 and packed in glass bottles (capacity: 240 cc, diameter: 65 mm, height: 120 mm), and, after the bottles were seamed, they were sterilized by heating at 121 °C for 20 minutes, thereby obtaining a hermetic container-packed, fat-containing beverage. The particle size

of the water-insoluble component of the homogenized fat-containing solution immediately after the homogenization was measured with a laser-type particle size distribution analyzer (SALD-1000), a product of Shimazu Co. The bottled beverage thus obtained was left standing at 25 °C, and the presence or absence of a cream layer was visually checked each week. The period required for the cream layer to have a thickness of 1 mm or more was investigated. The results are shown in Table 4.

[Amendment 11]

[Document to be amended] Specification

[Item to be amended] 0038

[Method of amendment] Conversion

[Details of the amendment]

[0038] [Table 4]

	实 施 例 (a)				比较例 (b)	
	5	6	7	8	3	4
昇温温度 (°C) (c)	60	60	80	60	60	60
均质化压力(kg/cm <sup>2</sup> ) *1	500	700	500	1000	700	150
最大粒子径 (μm) *2	0.92	0.68	0.87	0.54	0.68	1.89
冷却温度 (°C) (d)	20	20	40	20	50	20
乳化安定性 *3	10	12	16	18	2	1

Key: \*1) homogenizing pressure; \*2) largest particle size; \*3) emulsion stability; a) working examples; b) comparative examples; c) raised temperature; d) cooled temperature.

\*1 Nanomizer (a product of Nanomizer Co.) was used as the homogenizer.

\*2 The largest particle size of the water-insoluble component in the homogenized fat-containing solution immediately after the homogenization [measured with a laser-type particle size distribution analyzer (SALD-1000), a product of Shimadzu Co.]

\*3 Period (in weeks) until a cream layer was formed.

[Amendment 12]

[Document to be amended] Specification

[Item to be amended] 0039

[Method of amendment] Conversion

[Details of the amendment]

[0039] As seen from the results shown in Table 4, the thickness of the cream layers in all of the working examples was 0.2 mm or less for 10 weeks or thereabouts, thus indicating that the emulsified state was stable. In contrast, in Comparative Example 3, the homogenized fat-containing solution was not cooled sufficiently until the temperature became 40 °C or lower in the cooling step after the homogenization; as a consequence, the emulsion was destroyed in the sterilization step, and a cream layer was formed in only two weeks, thus rendering this example undesirable. In Comparative Example 4, because the particle size of the water-insoluble component of the homogenized fat-containing solution after the homogenization exceeded 1.0  $\mu\text{m}$ , the emulsion stability was poor, and a cream layer was formed in only one week, thus rendering this example undesirable.